

## PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.: 10/263,135 Confirmation No.: 2687  
Applicant(s): Fritzemeier et al.  
Filed: October 2, 2002  
Art Unit: 1742  
Examiner: Jenkins, Daniel J.  
Title: METHOD FOR PREPARING CRYOMILLED ALUMINUM ALLOYS  
AND COMPONENTS EXTRUDED AND FORGED THEREFROM

Docket No.: 038190/234776  
Customer No.: 00826

October 27, 2004

Commissioner for Patents  
P.O. Box 1450  
Alexandria, Virginia 22313-1450

## DECLARATION UNDER 37 C.F.R. § 1.131

Sir:

We, Daniel E. Matejczyk and Thomas J. Van Daam, hereby declare and state that:

1. We are inventors of the claimed invention of the above-identified U.S. Patent Application Serial No. 10/263,135.
2. At least as early as December 31, 2001, we reduced to practice our invention as described and claimed in the subject application, generally directed to a method of preparing cryomilled aluminum alloys and components extruded and forged therefrom.
3. Exhibit A (attached) is a copy of the "Nanophase Processing Yield/Scrap Tally" spreadsheet maintained by Raul Perez, working under the supervision of Dan Matejczyk. The spreadsheet shows that alloys were cryomilled as batch numbers NPCxx-05 and NPCxx-06 (portions of batch numbers have been redacted) at least as early as December 31, 2001. Cryomilling is a nanostructured material synthesis technique. Exhibit B (attached) shows handwritten lab notes, dated at least as early as December 31, 2001.

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which indicate the composition of the inert-gas-atomized starting powders that were weighed out and then cryomilled in an attritor. The composition of the metal powder, as indicated, was approximately 8 wt% Mg with the balance of the composition being approximately 92 wt% Al. 8 wt% Mg is well within the 1 atomic% to 11 atomic% range recited in the pending claims of the 10/263,135 application. As indicated on Exhibit A, both samples NPCxx-05 and NPCxx-06 were degassed in Impeller can #3, also designated as "Imp 3". Exhibit C (attached) is a copy of lab notebook pages that evidence the degassing and consolidation procedure applied to the contents of Impeller can #3. Exhibit D (attached) is a copy of hand-written notes showing the extrusion of the resulting consolidated billet from Impeller can #3 at least as early as December 31, 2001.

Exhibit E (attached) is a printout of laboratory results, dated at least as early as December 31, 2001, showing the Mg and Fe content of the extruded sample (Imp 3). Finally, Exhibit F (attached) is a copy of test results, dated at least as early as December 31, 2001, showing that the nitrogen content of the extruded sample (Imp 3) is above 0.3 wt%. The resultant nitrogen content indicated at least a 0.3 wt% increase in nitrogen content compared to the alloy powder prior to cryomilling. Dates, personal information, and other information not relevant to the substantiation of invention have been redacted from the copies included in the Exhibits. Although the dates have been redacted, each of Exhibits C, D, E, and F is dated at least as early as December 31, 2001.

4. During production of the Imp 3 sample described above, refractory material was not added to the alloy by anyone associated with production of the alloy. It would have been customary and regular practice to record any additions of refractory materials to the alloy in the cryomilling logs. The lack of any indication, in the cryomilling logs, that refractory material was added is positive evidence that no refractory material was added to the Imp 3 sample.

5. The attached exhibits evidence that, at least as early as December 31, 2001, we reduced to practice a cryomilled aluminum alloy by the steps of: providing a metal

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powder comprising 89 atomic% to 99 atomic% aluminum, 1 atomic% to 11 atomic% of a secondary metal selected from the group consisting of magnesium, lithium, silicon, titanium, zirconium, and combinations thereof; and processing the metal powder with a nanostructured material synthesis technique such that at least 0.3 weight% nitrogen was added to the metal; wherein refractory material was not added to the metal during processing. The exhibits further illustrate that we cryomilled such a powder, removed gaseous components from the cryomilled powder, consolidated the cryomilled powder into a metallic billet, and extruded the metallic billet, at least as early as December 31, 2001.

6. We hereby declare that all statements made herein of our own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issued thereon.

Daniel E. Matejczyk 10/27/04  
Daniel E. Matejczyk

Thomas J. Van Daam 10/27/04  
Thomas J. Van Daam

CLT01/4675634v3

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# Nanophase Processing Yield/Scrap Talley

Run No.	Date	Yield (Kg)	Yield %	Scrap (Kg)	Pwdr in Attrit	Attrit Run'g Tot	Comments
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Example 5

REDACTED

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NPC	05	19.625	98%	2.650	-2.275	12.590	R.H. 2%.
NPC	06	15.660	78%	3.010	+1.330	13.920	R.H. 4%. Packed impeller can #3 with 6/2 & 6/4 run.

REDACTED

NPC -05

## Powder Measure

5000g  
 5000g  
 5000g  
 2000g  


---

 17000g

} C.P. Al  
 Batch  
 #9 - 8035

in hopper

8<sup>25</sup> AM hopper on  
 8<sup>32</sup> 2N<sub>2</sub> Thru hopper  
 9<sup>00</sup> powder drop  
 9<sup>22</sup> @ 100 rpm  
 5<sup>10</sup> PM powder dump  
 5<sup>20</sup> in glove box

3000g } 50/50 Al-Mg  
 Batch #9 - 70245

40g Stearic Acid

R.H.  $\phi \rightarrow 2\%$ yield

3705g

4265g

4205g

4845g

2605g

---

19625Scrap

2650g

19625  
 2650  


---

 22275

20

2650g  
 3705g  
 4265g  
 4205g  
 4845g  
 2605g  


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 22275

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NPC -06

Powder Measure

5005 g	} C.P. A-l Batch #9 -8035
5105 g	
4890 g	
2000 g	
<u>17000 g</u>	

3000 g 50/50 Mg-A-P  
Batch #9 -70245

40g Stearic Acid

<u>7<sup>35</sup></u>	in hopper
<u>7<sup>45</sup></u>	hopper on
<u>7<sup>51</sup></u>	LN <sub>2</sub> Thru hopper
<u>8<sup>07</sup></u>	powder drop
<u>8<sup>14</sup></u>	@ 100 rpm
<u>4<sup>14</sup></u>	powder dump
<u>4<sup>23</sup></u>	in glove box

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<u>yield</u>	<u>scrap</u>	R.H. 4%
4250 g	3010 g	
4310 g		
4380 g		
<u>2720 g</u>		
15660 g		

# 42 TITLE Degas of Impeller can #3 PROJ. NO. Nanophase A1

## BOOK NO.

Work continued from Page

The powder was brought into the large glovebox and  
 welded shut on ~~1-inch~~ The can was packed on ~~33.785 kg~~ Can

8:20 AM Can was connected to vacuum pump.

10 ~~08:30~~

Time	Vac	I	Pwr	rpm	T <sub>probe</sub>	T <sub>c1</sub>	T <sub>c2</sub>	Further temp set	High limit
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8:33	$4 \times 10^{-2}$ (overshoot)	2.4	116W	38K	36°C				
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15 8:45	$2.05 \times 10^{-4}$	1.7	69W	38K	39°C				
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8:53	$9.98 \times 10^{-5}$	1.5	51W	38K	40°C				
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20 9:00	$6.85 \times 10^{-5}$	1.3	43W	38K	40°C	(chiller switched on)			
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9:40	$1.51 \times 10^{-5}$	1.1	32W	38K	34°C				
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25 8:50	$2.88 \times 10^{-5}$	1.0	30W	38K	33°C				
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9:20 • Vacuum level  $\sim 3.00 \times 10^{-5}$   $\Rightarrow$  There's a leak.  
 • Did a helium leak check, couldn't find a leak.  
 30 • Moving the O-ring results in slight vacuum changes.  $\Rightarrow$  Possible leak  
 • Did a 5 min nitrogen purge before exchanging ring  
 for a new one.

35 9:35 • open to high vacuum after changing O-ring and position  
 of O-ring

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TITLE Degas of Impeller Can #3

PROJECT NO. Nanophase AL 43  
BOOK NO.

Work continued from Page 42

Time	vac	I	Pwr	rpm	Temp
00:00	$3.73 \times 10^{-2}$ (Roughing)	24	111	38K	43°C
00:15	$1.23 \times 10^{-2}$ (Roughing)	20	84	38K	43°C
00:30	$5.08 \times 10^{-4}$	1.7	64	38K	42°C
00:45	$2.65 \times 10^{-4}$	1.4	50	38K	40°C
01:00	$1.89 \times 10^{-4}$	1.3	45	38K	39°C
01:05	$1.41 \times 10^{-4}$	1.3	41	38K	38°C
01:25	Helium check for leaks				
01:30	$1.09 \times 10^{-4}$	1.2	38	38K	37°C
01:35	$9.60 \times 10^{-5}$	1.2	37	38K	37°C
01:45	$8.16 \times 10^{-5}$	1.2	36	38K	37°C
01:50	$7.78 \times 10^{-5}$	1.1	35	38K	37°C

- completed helium check @ 11:40, no leaks detected  
- Vacuum is pumping too slow. N<sub>2</sub> back purge again (5min)  
and moved "O" again. Plus replaced "O" ring.

open to high vacuum once again

1.62 $\times 10^{-2}$ (Roughing)	2.1	92	38K	46°C
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1.82 $\times 10^{-4}$	2.0	81	38K	45°C
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a/ Roughing Pump @  $1.00 \times 10^{-2}$   
Work continued to Page 44

DATE

DATE

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Rail Perry

44 TITLE Degas of impeller can #3 PROJECT NO. Nanophase A1  
BOOK NO.

Work continued from Page 43						
Time	Vac	I	Pwr	rpm	T <sub>amp</sub>	
13:00	$4.78 \times 10^{-4}$	1.7	63W	38K	43°C	
13:15	$2.86 \times 10^{-4}$	1.5	53W	38K	42°C	
13:30	$1.76 \times 10^{-4}$	1.4	46W	38K	40°C	
13:45	$1.30 \times 10^{-4}$	1.3	41W	38K	40°C	
14:00	$9.88 \times 10^{-5}$	1.2	37W	38K	39°C	
14:15	$7.76 \times 10^{-5}$	1.1	35W	38K	38°C	
14:30	$6.33 \times 10^{-5}$	1.1	33W	38K	38°C	
14:45	$5.41 \times 10^{-5}$	1.1	32W	38K	37°C	
15:00	$4.91 \times 10^{-5}$	1.0	31W	38K	36°C	
15:15	$4.85 \times 10^{-5}$	1.0	31W	38K	36°C	
15:30	$4.35 \times 10^{-5}$	1.0	30W	38K	36°C	
15:45	$4.17 \times 10^{-5}$	1.0	30W	38K	35°C	
16:00	$3.97 \times 10^{-5}$	1.0	29W	38K	35°C	
16:15	$3.87 \times 10^{-5}$	1.0	29W	38K	35°C	
16:30	$3.75 \times 10^{-5}$	1.0	29W	38K	35°C	
N <sub>2</sub> Back Purge (5 min)						
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DATE						

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TITLE Degas of impeller can#3

PROJECT NO. Nanophase A1  
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Work continued from Page 44					
Time	Vac	I	Pwr	rpm	T <sub>imp</sub>
16:25	$1.89 \times 10^{-1}$ (Roughing)	5.4	346 W	38 K	42°C
17:00	$2.00 \times 10^{-2}$ (Roughing)	2.2	96 W	38 K	45°C
17:15	$4.55 \times 10^{-4}$	1.7	62 W	38 K	43°C
Helium check entire system for leaks					
7:20	$3.85 \times 10^{-5}$	1.0	30 W	38 K	33°C
<ul style="list-style-type: none"> <li>• N<sub>2</sub> back purge</li> <li>• Changed and moved O<sub>2</sub> ring again.</li> </ul>					
8:05	evacuation tube connected to high vacuum.				
8:30	$2.90 \times 10^{-5}$ (Roughing)	2.3	107 W	38 K	44°C
8:45	$6.78 \times 10^{-3}$ (Roughing)	1.9	75 W	38 K	43°C
9:00	$3.59 \times 10^{-3}$	1.5	52 W	38 K	40°C
9:45	$1.13 \times 10^{-4}$	1.2	39 W	38 K	38°C
10:15	$7.16 \times 10^{-5}$	1.2	34 W	38 K	37°C
12:30	$3.74 \times 10^{-5}$	1.1	32 W	38 K	42°C
Disconnected chiller					Work continued to Page 46
<small>SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA</small> <small>WORKMAN</small> <i>Rail Park</i> <small>DATE</small> <small>WITNESS</small> <small>DATE</small>					

46 TITLE Degas of impeller can #3 PROJECT NO. Nanophase A1  
BOOK NO.

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Time	Vac	I	Power	rpm	Temp
12:35	$3.73 \times 10^{-5}$	1.1	32W	38K	44°C

~~13:20 Helium detector used to find a small leak in side weld. The side weld was resealed in 2 spots.~~

Try to get helium detector to work. Not possible.  
14:30  $3.83 \times 10^{-5}$  1.0 30W 38K 45°C  
No improvement in vacuum reading  $\Rightarrow$  Definite leak

Mass spec Leak detector used to find a small leak in side weld. The side weld was resealed in 2 spots

13:26 Evacuation began

13:46	$2.55 \times 10^{-4}$	1.8	70W	38K	43°C
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14:00	$6.79 \times 10^{-5}$	1.2	40W	38K	38°C
-------	-----------------------	-----	-----	-----	------

14:45	$6.23 \times 10^{-5}$	1.0	29W	38K	34°C
-------	-----------------------	-----	-----	-----	------

18:35	$1.00 \times 10^{-6}$	1.0	28W	38K	34°C
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Good vacuum reading  $\Rightarrow$  no more leaks

9:40	$6.09 \times 10^{-7}$	1.1	30W	38K	33°C
------	-----------------------	-----	-----	-----	------

10:40	$6.05 \times 10^{-7}$	1.1	30W	38K	33°C
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N<sub>2</sub> Gas Purge (5 min)

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Paul King

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TITLE *Degas of impeller can #3*PROJECT NO. *Nanophase A1*  
BOOK NO.

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Time	U <sub>acc</sub>	I	Pwr	rpm	T <sub>amp</sub>	T <sub>ca</sub>	T <sub>ca2</sub>	Furnace temp/set	High Limit
11:30	$1.70 \times 10^{-4}$	1.4A	49W	38K	40°C				
13:00	$7.00 \times 10^{-6}$	1.1A	33W	38K	35°C				
14:00	$1.71 \times 10^{-6}$	1.0K	29W	38K	35°C				
14:30	$1.15 \times 10^{-6}$	1.0	29W	38K	35°C				
N <sub>2</sub> Gas back purge (5 min)									
15:45	$1.52 \times 10^{-4}$	1.4	49W	38K	42°C				
17:05	$1.10 \times 10^{-5}$	1.0	29W	38K	37°C				

HOT EVACUATION STARTED / 3 DAY RAMP TO 600°F

8:50	$3.97 \times 10^{-7}$	1.0	26W	38K	35°C	80	80	150/20	
9:55	<del>4.33</del>							<del>150/20</del> <sup>150/20</sup>	
9:10	$4.33 \times 10^{-7}$	1.0	27W	38K	35°C	118.2	118.8	150 hold/15min	163
9:25	$5.45 \times 10^{-7}$	1.0	27W	38K	35°C	120.4	118.6	158/25 (30m)	147
9:55	$7.59 \times 10^{-7}$	1.0	27	38	35	139.4	135.8	216/15min <sup>215</sup> hold	206
10:10	$9.41 \times 10^{-7}$	1.0	27	38	36	146.6	143.0	214/30min <sup>300</sup> ramp	205
10:40	$1.70 \times 10^{-6}$	1.0	27	38	36°C	185.0	179.2	300/300 <sup>60m</sup> hold	290
11:40	$7.48 \times 10^{-6}$	1.0	27	38	37°C	300	291	300/350 <sup>60m</sup> ramp	291
12:10								350 hold <sup>60m</sup>	

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*Dan Zimmer*

DATE

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48 TITLE Degas of impeller can #3

PROJECT NO. Nanophase A1  
BOOK NO.

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Time	Vac	I	Pwr	rpm	T <sub>comp</sub>	T <sub>c1</sub>	T <sub>c2</sub>	Turner rpm/set	High Volt
12:38	$1.77 \times 10^{-5}$	1.0A	27W	38K	37°C	258.6	258.6	350	341
13:10	$2.59 \times 10^{-5}$	1.0A	28W	38K	38°C	277.6	278.0	ramp to 375 30min	342
13:40	$2.98 \times 10^{-5}$	1.0A	28W	38K	38°C	293.6	289.4	375 hold 30m	367
14:25								ramp to 400 30min	
14:52	$3.24 \times 10^{-5}$	1.0A	28W	38K	38°C	309.8	306.2	ramp	376
15:55	$3.15 \times 10^{-5}$	1.0A	28W	38K	38°C	321.8	318.4	400 hold	394
16:00	$2.86 \times 10^{-5}$	1.0A	28W	38K	37°C	337.8	334.8	400 hold	395
17:00	$2.46 \times 10^{-5}$	1.0A	28W	38K	36°C	346.2	343.8	400 hold	395
18:20	$8.69 \times 10^{-6}$	1.0	28W	38K	33°C	384.2	382.8	400 hold	399
18:30	$8.71 \times 10^{-6}$	1.0	28W	38K	33°C	384.2	383.0	450/30	399
19:45	$8.80 \times 10^{-6}$	1.0	29W	38K	32°C	385.8	384.6	450/30	445
19:00	$8.91 \times 10^{-6}$	1.0	29W	38K	32°C	390.6	389.0	450 hold	444
19:15	$9.46 \times 10^{-6}$	1.0	29W	38K	32°C	394.4	392.6	450 hold	448
19:30	$1.01 \times 10^{-5}$	1.0	29W	38K	32°C	397.0	395.0	500/30	448
19:45	$1.10 \times 10^{-5}$	1.0	29W	38K	32°C	405.2	403.2	500/30	475

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Laila Perry

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TITLE impeller can #3

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Time	Vac	I	R/r	rpm	Temp	TC <sub>1</sub>	TC <sub>2</sub>	Furnace Temp/Set	High Limit
5 10:00	$1.22 \times 10^{-5}$	1.0	29	38 K	32°C	415.2	413.2	500/hold	497
10:15	$1.42 \times 10^{-5}$	1.0	29 W	38 K	31°C	421.4	419.4	500/hold	495
10:30	$1.67 \times 10^{-5}$	1.0	29 W	38 K	31°C	426.2	424.2	525/30	495
10 10:45	$1.94 \times 10^{-5}$	1.0	29 W	38 K	31°C	432.4	430.2	525/30	508
11:00	$2.42 \times 10^{-5}$	1.0	29 W	38 K	31°C	441.8	439.6	525/hold	521
15 11:15	$2.78 \times 10^{-5}$	1.1	29 W	38 K	31°C	445.4	443.6	525/hold	519
11:30	$3.19 \times 10^{-5}$	1.1	29 W	38 K	31°C	449.4	447.4	525/hold	520
11:45	$3.63 \times 10^{-5}$	1.1	29 W	38 K	31°C	453.2	451.4	525/hold	525
20 12:00	$4.21 \times 10^{-5}$	1.1	30 W	38 K	31°C	463.8	462.0	525/hold	565
12:15	$4.69 \times 10^{-5}$	1.1	30 W	38 K	31°C	467.0	465.4	525/hold	565
25 12:30	$5.69 \times 10^{-5}$	1.1	30 W	38 K	31°C	470.6	469.0	550/30	563
12:45	$6.28 \times 10^{-5}$	1.1	31 W	38 K	31°C	476.6	475.2	550/30	580
13:00	$7.27 \times 10^{-5}$	1.1	31 W	38 K	30°C	484.4	483.4	550/hold	596
30 13:15	$8.33 \times 10^{-5}$	1.1	31 W	38 K	30°C	490.0	488.6	550/hold	565
13:30	$9.88 \times 10^{-5}$	1.1	32 W	38 K	30°C	493.6	492.2	550/hold	564
35 13:45	$1.19 \times 10^{-4}$	1.2	33 W	38 K	31°C	496.8	495.4	550/hold	563

SCIENTIFIC BINDER PRODUCTIONS CHICAGO 60665 MADE IN USA

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50 TITLE impeller can #3

PROJECT NO.

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Time	Uac	I	Power	RTM	Pump	Le	TCZ	Furnace temperature	High limit
14:00	$1.44 \times 10^{-4}$	1.2	33 W	38 K	31°C	499.2	497.8	550/hold	563
14:15	$1.74 \times 10^{-4}$	1.2	34 W	38 K	31°C	506.2	500.0	550/hold	563
14:30	$2.07 \times 10^{-4}$	1.3	35 W	38 K	31°C	507.0	501.8	550/hold	563
14:45	$2.48 \times 10^{-4}$	1.3	36 W	38 K	31°C	505.0	503.4	550/hold	562
15:00	$2.91 \times 10^{-4}$	1.3	39 W	38 K	32°C	506.2	504.8	550/hold	562
15:15	$3.36 \times 10^{-4}$	1.4	41 W	38 K	32°C	507.4	506.2	550/hold	562
15:30	$4.03 \times 10^{-4}$	1.5	45 W	38 K	32°C	508.8	507.6	550/hold	562
15:45	$4.72 \times 10^{-4}$	1.7	54 W	38 K	33°C	510.2	509.8	550/hold	561
16:00	$5.04 \times 10^{-4}$	1.8	62 W	38 K	33°C	510.8	509.6	550/hold	561
16:15	$4.46 \times 10^{-4}$	2.0	73 W	38 K	34°C	511.8	510.2	550/hold	561
16:30	$6.29 \times 10^{-4}$	2.2	85 W	38 K	35°C	512.6	511.2	550/hold	561
16:45	$8.80 \times 10^{-4}$	2.4	99 W	38 K	36°C	513.4	511.8	550/hold	561
17:00		2.5	107 W	38 K	37°C	514.0	512.6		560
17:15		2.6	119 W	38 K	38°C	514.8	513.2	550/hold	560
17:30		2.7	121 W	38 K	39°C	515.4	513.8		561
17:45									
18:00									

EMERY PRODUCTIONS CHICAGO 60608 MADE IN USA

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DATE

DATE

TITLE

impeller can #3

PROJECT NO.

51

BOOK NO.

Work continued from Page 50

Time	Vac	I	Amr	rpm	T <sub>amp</sub>	T <sub>c1</sub>	T <sub>c2</sub>	Furnace Temp/Set	H <sub>2</sub> O limit
17:45		2.7	122 W	38 K	39°C	514.0	514.4	550/hold	560
		Roughing Pump $3.98 \times 10^{-1}$ → PEAK							
18:00		2.7	120 W	38 K	40°C	514.6	515.0	550/hold	560
		Roughing Pump $3.84 \times 10^{-1}$							
18:15		2.6	119 W	38 K	40°C	517.0	517.4	550/hold	560
		Roughing Pump $3.57 \times 10^{-1}$							
18:30		2.5	112 W	38 K	40°C	517.4	515.8	550/hold	560
		Roughing Pump $3.12 \times 10^{-1}$							
18:45		2.3	105 W	38 K	40°C	517.8	516.2	550/hold	560
		Roughing Pump $2.65 \times 10^{-1}$							

8:45	$7.59 \times 10^{-5}$	1.1	33 W	38 K	33°C	522.2	520.8	575/30	579
9:00	$7.52 \times 10^{-5}$	1.1	32 W	38 K	33°C	525.6	524.4	575/30	575
9:15	$7.70 \times 10^{-5}$	1.1	32 W	38 K	33°C	529.8	529.4	575/hold	589
9:45	$8.21 \times 10^{-5}$	1.1	32 W	38 K	34°C	535.6	534.4	575/hold	587
10:19	$8.83 \times 10^{-5}$	1.1	33 W	38 K	34°C	538.4	537.2	575/hold	587

Work continued to Page 52

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

DATE

DATE

DATE

WITNESS

52 TITLE

impeller can #3

PROJECT NO.

BOOK NO.

Work continued from Page 51

	VAC	I	Pwr	rpm	T <sub>mag</sub>	T <sub>c1</sub>	T <sub>c2</sub>	Humidity Temp/Sec	High Limit
10:45	7.99 x 10 <sup>-5</sup>	1.1	33W	38k	35°C	539.6	541	610/30	587
11:15	9.90 x 10 <sup>-5</sup>	1.1	33W	38k	35°C	554.4	553.4	610/hold	630
13:00	1.52 x 10 <sup>-4</sup>	1.2	37	38k	37°C	567.6	568.4	610/hold	623
	roughing pump 6.86 x 10 <sup>-3</sup>								
14:15	1.69 x 10 <sup>-4</sup>	1.3	37	38k	38°C	573.6	572.4	625/30	621
	roughing pump 8.14 x 10 <sup>-3</sup>								
15:10	1.70 x 10 <sup>-4</sup>	1.3	38	38k	38°C	583.4	582.2	625/Local	638
	roughing pump 9.22 x 10 <sup>-3</sup>								
16:00	1.72 x 10 <sup>-4</sup>	1.3	38	38k	38°C	587.2	586.0	625/Local	637
17:00	1.59 x 10 <sup>-4</sup>	1.3	37	38k	38°C	587.8	588.8	625/Local	636
	roughing pump 7.50 x 10 <sup>-3</sup>								
09:00	2.49 x 10 <sup>-5</sup>	1.0	28W	38k	38°C	594.0	592.4	625/Local	635
09:10	2.52 x 10 <sup>-5</sup>	1.0	28W	38k	38°C	594.0	592.6	632/Local	635
09:30	2.55 x 10 <sup>-5</sup>	1.0	28W	38k	38°C	596.8	595.2	632/Local	643
10:30	2.57 x 10 <sup>-5</sup>	1.0	28	38k	38°C	598.6	597.2	632/Local	643
12:00	2.47 x 10 <sup>-5</sup>	1.0	28	38	38°C	599.6	598.2	632/Local	642
15:00	2.20 x 10 <sup>-5</sup>	1.0	27	38	37°C	600.6	599.2	632/Local	642
17:38	1.93 x 10 <sup>-5</sup>	1.0	28W	38k	36°C	600.8	599.6	632/Local	642

BINDER PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page 53

DATE

DATE

WITNESS TO AND UNDERSTOOD BY

DATE

WITNESS

DATE

53

TITLE Impeller 'can' #3

PROJECT NO.

BOOK NO.

Work continued from Page 52

Time	Nac	I	Pwr	ram	Temp	T <sub>c1</sub>	T <sub>c2</sub>	Furnace Temp/Set	Atch limit
9:30	$9.60 \times 10^{-6}$	1.0	27 W	38 K	34°C	600.4	599.0	632/loc	642
9:30	$2.85 \times 10^{-6}$	1.0	28 W	38 K	32°C	600.0	598.6	632/loc	641
9:41	$2.85 \times 10^{-6}$	1.0	28 W	38 K	32°C	600.2	598.8	632/loc	641
• Shut Furnace off and chiller									
9:40	$1.33 \times 10^{-6}$	1.0	27 W	38 K	33°C	470.6	475.6		
9:40	$7.78 \times 10^{-7}$	1.0	26 W	38 K	34°C	444.4	445.8		
9:40	$2.81 \times 10^{-7}$	0.9	26 W	38 K	34°C	400.6	402.2		
9:45	$1.45 \times 10^{-7}$	0.9	26 W	38 K	35°C	372.6	374.6		
9:50	$6.05 \times 10^{-8}$	0.9	25 W	38 K	35°C	311.6	312.2		
9:50	$5.36 \times 10^{-8}$	0.9	25 W	38 K	35°C	275.2	250.6		
• Thermo couples disconnected									
• Tube crimped. (ultimate vacuum $9.50 \times 10^{-9}$ a HODMAN)									
9:55	$5.12 \times 10^{-8}$	0.9	25 W	38 K	35°C				
• Tube welded									
• Vacuum level jumped all around and peaked @ $7.01 \times 10^{-8}$									
9:58	$5.12 \times 10^{-8}$								

Work continued to Page 54

DATE

DATE

DATE

WITNESS

PAGE 27/25 / BCD AT 10/7/2005 3:14:06 PM (Eastern Daylight Time) \* SVR:USPTO-EFXXF-6/25 \* DNIS:2738300 \* CSID:7043316090 \* DURATION (mm:ss):06:56

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54 TITLE

impeller can #3

PROJECT NO. Nanophase A1  
BOOK NO.

Work continued from Page 53

Final Mass of can 33.665 Kg  
Tube cut off 0.045 Kg  
33.710

Initial can Mass 33.765 Kg

\* Tube was cut 3 times.

Can was shipped out on  
8/22

SCIENTIFIC BINDERY PRODUCTIONS CHICAGO 60605 MADE IN USA

Work continued to Page

SIGNATURE

D. M. Z. Guntt

DATE

RECEIVED TO AND UNDERSTOOD BY

DATE

WITNESS

DATE

IMP1

4 INDUERS (9,10,11)(12)

IMP2

SHIPPED IMP3 &amp; IMP4 ON AUG 20 (MID SEPT. EXTRUSION)

\* FINAL FURNACE SETPOINT WAS 371°F

IMP2 LOADED INTO FURNACE @ 8:00am.

TIME	BILLET TEMP T/C #2	FURNACE TEMP #3
7:00am		
7:15am	389.2	388.2
7:20am	389.1	390.2
7:40am	389.3	392.8
8:45am	380.8	386.2
9:05am	377.1	382.3
9:30am	372.2	378.5
	371	378.0
9:46	370	375
		374

3 1/2 POUNDS BILLET LOADED @ 10:30 PM  
1:30 PM + 10:30 PM

REDACTED

BREAK THROUGHT F<sub>IND</sub> = 4237 TONS

IMP 3 &amp; 4

FURNACE LOAD 3:45am @ 400°F  
200°F @ 4:10am  
350°F @ 6:00am  
380°F @ 7:00am

	BILLET TEMPS		
	T/C #2	T/C #3	
7:00am			FURNACE SETPOINT DROPPED FROM 410 → 400
7:15am	389.2	388.2	BILLET 4 9-30-98-IMP4 [TORN JACKET]
	389.1	390.2	400.3
	389.3	392.8	400.8 ... FROM 400 → 376°F
7:20am			399.4
7:40am	389.3	391.6	386.2 ... FROM 376 → 369°F
8:45am	380.8	386.2	... 8:50 369 → 364
9:05am	377.1	382.3	381.0
9:30am	372.2	378.5	376.5 364 → 368
	371	378.0	375
9:46	370	375	LIVER 634°F
		374	(377 DIE 200 → 400°F) @ 9:30

PULLED FIRST.

950 FIRST RUN

10:30

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THE BOEING COMPANY  
Rocketdyne Propulsion  
And Power System  
MATERIALS APPLICATION  
PROCESS  
LABORATORY OPERATIONS

DATE  
G.O.  
61918  
PROGRAM NAME  
Nanophase Aluminum  
MATERIAL  
Aluminum Alloy  
SUBMITTED BY  
R. Perez

REFERENCE  
EWR#936081

REPORT No.  
9811-4  
QUANTITY  
2  
SPECIFICATION

DEPT.  
912

LOCATION  
CANOGA

PHONE  
3958

## TEST REPORT

## LABORATORY TEST RESULTS

SAMPLE	Mg %/wt	Iron %/wt
9/30 IMP3	8.8	0.1
9/30 IMP4	8.6	0.1

Log No. 9811-4  
Charge No. 24410-61918-98100  
Notebook Page No. 2054-24

W. Ho

ANALYST

DATE

A. Appti (W. Ho)

APPROVED

DATE



**LECO CORPORATION**  
 3000 Lakeview Avenue  
 St. Joseph, Michigan 49085-2398

Work Order No: 12538  
 Classification: 8  
 POWO No: NR98439023  
 Customer No: 2593  
 Account No: 109485

## CUSTOMER SERVICE ANALYSIS REPORT

Customer: BOEING NORTH AMERICAN, INC.  
 ROCKETDYNE DIVISION/633 CANOGA AVE.  
 CANOGA PARK CA 91303

Attention: SONYA REID Dept. 917, MC BA71

The following is our laboratory analysis report on samples submitted by your company.

Any additional details of this analysis report can be obtained from the Technical Services Laboratory.  
 Call 616-982-2277.

*NOTE: We request that all samples submitted to the LECO® Technical Services Laboratory be accompanied by a Material Safety Data Sheet (MSDS) describing each material's characteristics. This information permits proper handling and storage.*

Results for Sample: 63681 IMP3 A17.5%Mg

Instrument: CS444

Standard: NIST 125b @ 0.028% C

Remarks: Preheated Crucible 528-018, Preheated Lid 528-043, ~1 g LecoCel II HP 502-173, ~1 g Iron Chip 502-231

Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

% Sulfur	% Carbon
	0.152
	0.151
	0.148

Instrument: RH402

Standard: Hydrogen Gas Dose

Remarks: 769-781 Crucible

Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

Hydrogen ppm	Bulk ppm	Surface ppm
43.09	42.78	0.311

Instrument: TC435

Standard: LECO Std. 501-544 @ 0.0107% O, 0.0071% N (J0215-2)

Remarks: 782-720 Crucible, 1 g Nickel Basket 502-344, 0.05 g Graphite Powder 501-073

Preparation: Abraded with a file, sectioned with a shear, rinsed in acetone and dried with warm air.

% Nitrogen	% Oxygen
0.383	0.239
0.381	0.235

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